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SELECTIVELY THERMALIZED SPUTTERING FOR THE DIRECT
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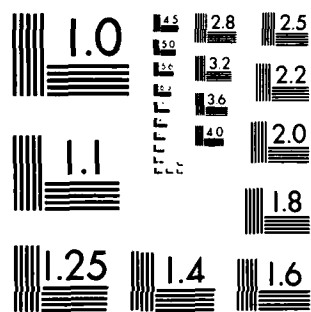
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SELECTIVELY THERMALIZED SPUTTERING
FOR THE DIRECT SYNTHESIS OF Sm-Co
AND Sm-Fe FERROMAGNETIC PHASES

Final Technical Report

Dr. F. J. Cadieu

22 March 1983

U. S. Army Research Office

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TABLE OF CONTENTS

Report Documentaion Form DD 1473	2,3
Table of Contents	4
List of Illustrations	5
Abstract	7
Introduction	8
Basic Experimental Procedures	10
Major Results and Conclusions	15
Research in Progress	27
References	28
Contract Publications	29
Participating Scientific Personnel	30

LIST OF ILLUSTRATIONS

	page
Fig. 1. Calculated thermalization of sputtered atoms in 150 mTorr of Ar for a target to substrate distance of 2 cm. Since Fe and Co have similar masses their thermalization profiles will be similar.	12
Fig. 2. Cu diffractometer trace for directly synthesized Sm-Co film with 16.7 at. % Sm. This sample had an oxygen content of 9.7 at. %.	17
Fig. 3. Cu diffractometer trace for directly synthesized Sm-Co film with 16.7 at. % Sm. This sample had an oxygen content of 6.5 at. %.	17
Fig. 4. Cu diffractometer trace for directly synthesized Sm-Co film with 16.7 at. % Sm. This sample had an oxygen content of 1.4 at. %.	17
Fig. 5. Film magnetization of Sm-Co 1:5 (16.7 at. % Sm) versus applied field; substrate temperature = 700 C, sputtering gas = 150 mTorr Ar, oxygen level = 6.5 at. %.	18
Fig. 6. Film magnetization versus parallel and perpendicular applied fields. This is the same sample as in Fig. 5.	18
Fig. 7. Parallel to film plane and perpendicular hysteresis loops for a Sm-Co 1:5 sample synthesized onto 650 C substrate without any applied magnetic field during the sputtering, oxygen level = 1.4 at. %.	19
Fig. 8. X-Ray diffraction trace for the sample of Fig. 7. The (100) and (300) reflections are present off the ends of this trace. $I(200)/I(110) = 20:1$.	19
Fig. 9. Magnetization as a function of inplane angle for a magnetometer field of 15 kOe. During the sputter synthesis there was a field of 1.75 kOe applied inplane at 0 deg. reference angle.	21
Fig. 10. Parallel to film plane in direction of field applied during sputtering hysteresis loop. This sample was sputtered with inplane field of 1.75 kOe at 0 deg.	21
Fig. 11. Inplane hysteresis loop for sample of Fig. 10, but at 90 degrees to field applied during sputtering.	23
Fig. 12. X-Ray diffraction trace for sample of Fig. 10,11.	23

	page
Fig. 13. Parallel to film plane and perpendicular to film plane hysteresis loops for Sm-Fe (16.7 at. % Sm) plus oxygen system sample. This composition would correspond to a 1:5 compound.	26
Fig. 14. Sm-Fe plus oxygen system showing maximum in remanent induction and coercivity at a composition corresponding to a 1:5 Sm-Fe metastable compound. The out of plane moment is larger than in plane moment for an applied field of 14 kOe. Magnetic measurements made at Grumman Aerospace Corp.	26

TABLE 1. Inplanar Magnetization	20
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ABSTRACT

Ferromagnetic films of the Sm-Co and Sm-Fe systems have been synthesized by sputtering onto substrates at temperatures above 600 C so that the deposit is directly crystallized upon deposition. For the directly synthesized phases, high sputtering gas pressures were used so that the sputtered atoms transferred excess momentum to the sputtering gas atoms before arriving at the substrates. The object has been to promote the growth of possible metastable phases and to allow preferred orientation effects to be present in the films. Directly synthesized Sm-Co 1:5 films grown by these methods have been observed to be highly textured with the (200) orientation dominant for low oxygen levels and then switching to a (110) texture for oxygen levels greater than 5 atomic %. The c-axis of the 1-5 phase is rigidly aligned into the film plane. The oxygen to film texture correlation exists for substrate temperatures from $T = 585$ to 600 C. Samples of the Sm-Co 1:5 compound have been directly crystallized by sputtering onto heated substrates with and without an applied inplane field of 1.75 kOe during the sputter deposition. The inplane field during sputtering has been used to preferentially align the c-axis within the film plane. Square flat topped hysteresis loops have been observed for samples sputtered with an applied inplane field. Values of M remanence to M (14 kOe) = 0.97 have been observed. In the Sm-Fe films with 6.5 atomic % oxygen, a new metastable phase has been observed at a composition corresponding to a 1-5 compound. This oxygen stabilized metastable phase is not present in Sm-Fe films made with lower amounts of oxygen incorporation.

SELECTIVELY THERMALIZED SPUTTERING FOR THE DIRECT SYNTHESIS OF

Sm-Co AND Sm-Fe FERROMAGNETIC PHASES

INTRODUCTION

Ferromagnetic films of the Sm-Co and Sm-Fe systems have been synthesized by RF sputtering onto heated substrates so that the samples are directly crystallized upon deposition. The sputtering target arrangement was such that a range of compositions was deposited along the length of the substrates. The resulting Sm to Co ratio, for example, then varies systematically along the length of the 5 cm long substrates with separate subregions having a composition resolution on the order of 1 atomic %. Samples with slightly different compositions, but with identical preparation conditions, could then be analyzed by observing small subregions along the length of a single substrate. The sputtering gas pressures used were sufficiently high so that the sputtered atoms were thermalized by collisions with the sputtering gas atoms before arriving at the substrates. The substrate temperatures were maintained from 600 to 800 C during deposition. These substrate temperatures were high enough so that the deposited films were directly crystallized as deposited, hence the term direct synthesis.

The use of selectively thermalized sputtering in which the sputtered atoms are thermalized to the substrate temperature as they arrive at the substrate (1,2) allows a delicate and or metastable phase to be replicated by the subsequently arriving sputtered atoms. In addition, differing amounts of oxygen have been used during sputtering with dramatic effects

observed in the crystallographic and associated magnetic properties.

Early preliminary results (3) showed the c-axis for the Sm-Co 1-5 compound to be rigidly aligned into the film plane of the sputtered samples. In these early results, however, the full moments expected were not observed. Subsequent work showed the crystallographic texture observed and the associated magnetic properties to be a strong function of the amount of oxygen incorporated into the films during the sputter synthesis (1). For the Sm-Co 1-5 composition, there was a well defined change in the resulting crystallographic orientation of the films which depended upon the amount of oxygen incorporated into the films. For oxygen concentrations below 5 atomic % the (200) texture became successively dominant. For oxygen concentrations greater than 5 atomic % the (110) texture became dominant. In either of these textures the c-axis tended to lie in or near the plane of the substrates.

For the Sm-Fe system, oxygen incorporated into the films during the growth process had the dramatic effect of stabilizing a new phase at the composition corresponding to a 1-5 compound (1). The diffraction lines associated with this compound were not present when the oxygen level was reduced to about 1 atomic % oxygen. This result is consistent with the results of Buschow (4) for the bulk Sm-Fe system in that only the 1-2, 1-3 and 2-17 phases are present. We have however been able to stabilize a metastable Sm-Fe compound which we have tentatively identified as an oxygen stabilized 1-5 compound.

In our most recent work the Sm-Co system has been synthesized with an applied magnetic field of 1.75 kOe in the plane of the substrates during the sputter synthesis process.(5) Results to date with this new

technique have mostly been confined to the Sm-Co 1-5 phase. The purpose of the magnetic field applied during the synthesis process is to see if the c-axis of the crystalline films can be preferentially aligned in the direction of the magnetic field applied during synthesis. Highly square flat topped hysteresis loops for the Sm-Co 1-5 compound have been observed in thick film samples directly synthesized by this combination of techniques. These square loop properties have been obtained on directly synthesized samples without the need for any subsequent annealing or step aging.(5)

BASIC EXPERIMENTAL PROCEDURES

Samples have been made by RF sputtering in an all stainless steel chamber with a base pressure in the low nanoTorr range. This chamber was pumped by inert gas ion pumps and a titanium sublimation pump. During sputtering, the titanium sublimation cryopanel was cooled by liquid nitrogen to help maintain a low contamination atmosphere. A custom sputtering electrode was used so that sputtering occurred from three colinear targets simultaneously, which we term trisputtering. By varying the target compositions individually, a range of compositions could then be created in the deposited films so that a systematic gradient occurred in one or more components along the length of the substrates. By this procedure samples with a systematic composition difference in one or more components on the order of 1 atomic % could then be studied by observing separate subregions of a single substrate. The substrates were polished polycrystalline aluminum oxide measuring 1.8 cm by 5.0 cm by 0.06 cm. Substrate temperatures during deposition could be varied from room temperature to temperatures up to 1000 C. Generally

substrate temperatures above 600 C were used so that the sputtered material was directly crystallized onto the substrate during deposition. The samples were not subsequently annealed or heat treated in any way before the magnetic properties were measured. In certain test cases samples were first made amorphous by sputtering onto low temperature substrates and then subsequently crystallized by annealing.

Samples have been sputtered with and without an inplane magnetic field of 1.75 kOe applied in the plane of the substrates during deposition. The field was applied during sputtering in the substrate plane and perpendicular to the long axis of the substrates. This field was thus also perpendicular to the composition gradient along the length of a substrate.

The sputtering gas pressures used have been high enough so that most of the sputtered atoms were thermalized through collisions with the sputtering gas atoms as they arrived at the substrate. The sputtered atoms were predominately neutral atoms with an asymmetric energy distribution centered at approximately 5-10 eV and extending up to about 100 eV. In Fig. 1 we show a calculated change in sputtered atom energy as the sputtered atoms progress from the targets to the substrate. A sputtering gas pressure of 150 mTorr Ar has been used for most of the films reported. As indicated in Fig. 1 this pressure should be sufficient to lower the sputtered atom temperature to that of the substrate before arrival at the substrate. Some of the high energy tail of the sputtered atom momentum distribution was still probably not fully thermalized at this pressure. A more optimum thermalization could be obtained by the use of a two component sputtering gas in which Ar was used to thermalize the lighter

3-d element, either Fe or Co, and an admixture of Xe used to selectively thermalize the more massive Sm atoms. Currently a system for bleeding Xe gas into the sputtering system is being installed.

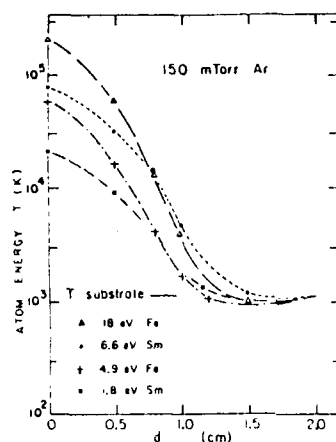


Fig. 1. Calculated thermalization of sputtered atoms in 150 mTorr of Ar for a target to substrate distance of 2 cm. Since Fe and Co have similar masses their thermalization profiles will be similar.

Variations in the Sm to Co atomic ratio along the length of a substrate were measured directly on the sputtered films by quantitative x-ray fluorescence. These composition measurements were calibrated by preparing pseudo thin films by dissolving known ratios of the elements in acids and drying onto filter papers. The crystal phases present and the extent of crystallinity versus amorphous component were determined by diffractometer tracings on subregions of a substrate using Cu radiation. A Si(Li) solid state x-ray detector was used which allowed the complete discrimination

against Co fluorescence being accepted as Cu diffraction counts. With this detector diffraction traces could also be obtained so that only Cu K Beta was accepted. This detector allowed flat, low background diffractometer data to be collected so that an amorphous hump could be clearly seen if present. During the course of these studies the x-ray diffraction analysis was changed so that the patterns could be directly collected and analyzed by a small computer. This automation allowed a far greater number of samples to be analyzed at a minimum of man hours.

Most samples were sputtered to a thickness of 2-4 micrometers. The thickness of various films has been determined by SEM measurements and by the weighing of substrates before and after deposition. Relative x-ray fluorescence measurements and beta back scattering has also been used to measure film thicknesses.

The oxygen composition of the sputtered films was determined by measuring the relative oxygen to cobalt and or iron Auger intensity ratio with a Varian high resolution cylindrical mirror analyzer. An argon ion gun was used during analysis to eliminate surface contamination. The total atomic percentage of oxygen was then calculated utilizing the atomic ratio of 3-d element to Sm as determined by the x-ray fluorescence.

After various regions of a substrate were characterized by x-ray fluorescence and diffraction measurements small subregions of a substrate were sectioned out for magnetometer measurements. Subregions 0.4 cm square were normally used so that the sample could be rotated in a vibrating sample magnetometer probe. The magnetization of the attached

aluminum oxide substrate was characterized and found to be diamagnetic with an isotropic magnetic susceptibility of $\chi = -0.4 \pm 0.1 \times 10^{-6}$ emu/g. For the sample size used, the substrate gave rise to a maximum magnetization of $M \approx -2 \times 10^{-4}$ emu at $H = 14$ kOe. This was less than 1% of the magnetization of the Sm-Co and Sm-Fe films studied. Consequently the contribution of the substrate to the observed magnetization was neglected in the analysis. Most of the magnetometer measurements were made at the Grumman Aerospace Corporation using a Princeton Applied Research vibrating sample magnetometer with applied fields to 14 kOe. Certain measurements were also made at Fort Monmouth with applied fields up to 15 kOe. On selected samples high field measurements to 150 kOe were also obtained at the Francis Bitter National Magnet Laboratory.

MAJOR RESULTS AND CONCLUSIONS

Approximately 150 substrates for the Sm-Co and Sm-Fe systems have been sputtered onto heated substrates under the conditions of thermalized sputtering so as to promote the direct synthesis of crystalline phases. Most of the magnetic measurements we have been able to perform have been on the 1:5 composition region of the Sm-Co system. Measurements of samples of the Sm-Fe system will be discussed after conclusions pertaining to the Sm-Co 1:5 composition region have been discussed.

Directly synthesized sputtered films of the Sm-Co 1:5 compound had the c-axis rigidly aligned into the plane of the substrate. Films sputtered in the absence of any inplane field during sputtering evidenced a random distribution of the c-axis within the film plane. The resulting film texturing and magnetic properties were a strong function of the amount of oxygen incorporated into the films during sputtering. Samples with an oxygen content of less than 5 atomic % showed a predominantly (200) texture. Samples which had an oxygen content of greater than 5 atomic % showed a progressively (110) texture dominance. This is illustrated in Fig.'s 2-4. Samples sputtered onto heated substrates at $T = 585$ to 800 C showed a correlation of film texture to oxygen content. Samples sputtered onto slightly lower temperature substrates showed a (110) dominant texture which is believed due to argon incorporation into the films at the lower substrate temperatures. The hysteresis loops of samples with oxygen contents of greater than about 5 atomic % generally showed a change of slope upon entering the second quadrant. This is illustrated in Fig.'s 5 and 6. For a magnetizing field of 150 kOe the saturation moment was 105 emu/g.

This is the expected bulk value. The moment for fields up to 14 kOe applied perpendicular to the film plane is also shown. This loop is highly skewed as expected for the c-axis being rigidly aligned into the film plane.

In Fig. 7 a hysteresis loop is illustrated for a Sm-Co 1:5 phase is illustrated for a 1.4 atomic % oxygen sample. The loop is nearly square in this case but still has an appreciable slope to the top and bottom segments. An x-ray diffraction trace for this sample is shown in Fig. 8. The intensity ratio of the (200) to (110) reflection in this case is 20:1. No c-axis reflections are present. Also present are the (100) and (300) reflections.

In an effort to align the c-axis of the Sm-Co 1:5 compound preferentially within the film plane, this phase was directly synthesized by thermalized sputtering with a magnetic field of 1.75 kOe applied in the substrate plane during sputter deposition. This was only partially successful. In Fig. 9 is shown the moment as a function of inplanar angle with respect to the direction of the field applied during sputtering. This sample was rotated in a constant magnetometer field of 15 kOe applied in the film plane. Although only limited preferential alignment of the c-axis within the film plane was achieved, the hysteresis loops of films with low oxygen sputtered with an applied inplane field during sputtering were flat topped and square. In Table 1 certain parameters are indicated for a 1:5 Sm-Co film sputtered with an applied inplane field during the sputter synthesis.

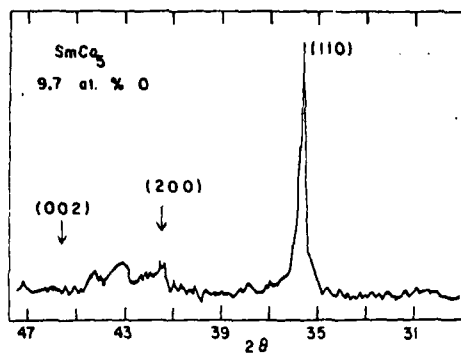


Fig. 2. Cu diffractometer trace for directly synthesized Sm-Co film with 16.7 at. % Sm. This sample had an oxygen content of 9.7 at. %.

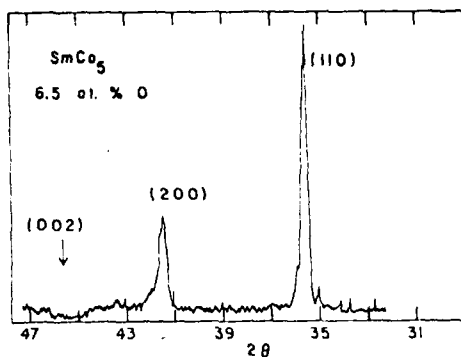


Fig. 3. Cu diffractometer trace for directly synthesized Sm-Co film with 16.7 at. % Sm. This sample had an oxygen content of 6.5 at. %.

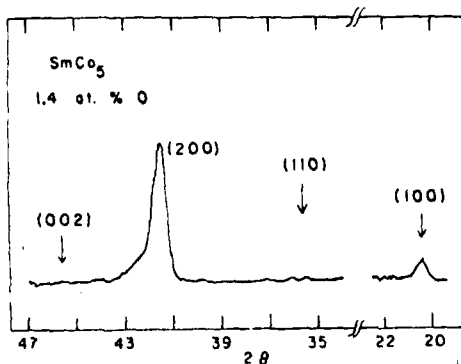


Fig. 4. Cu diffractometer trace for directly synthesized Sm-Co film with 16.7 at. % Sm. This sample had an oxygen content of 1.4 at. %.

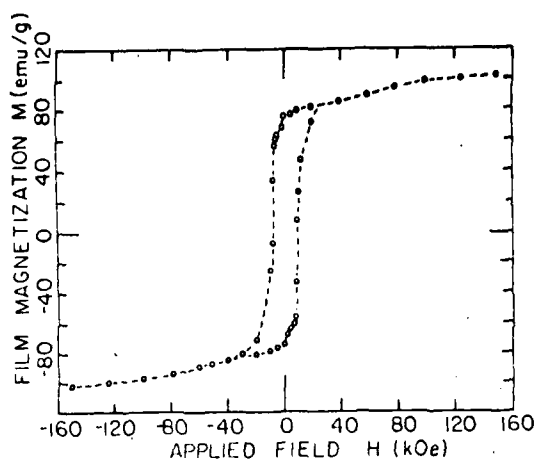


Fig. 5. Film magnetization of Sm-Co 1:5 (16.7 at. % Sm) versus applied field; substrate temperature = 700 C, sputtering gas = 150 mTorr Ar, oxygen level = 6.5 at. %.

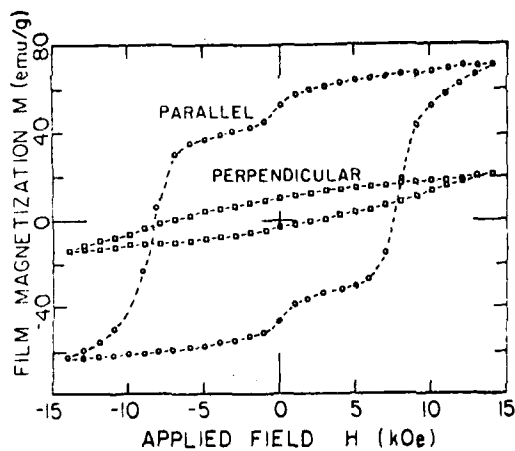


Fig. 6. Film magnetization versus parallel and perpendicular applied fields. This is the same sample as in Fig. 5.

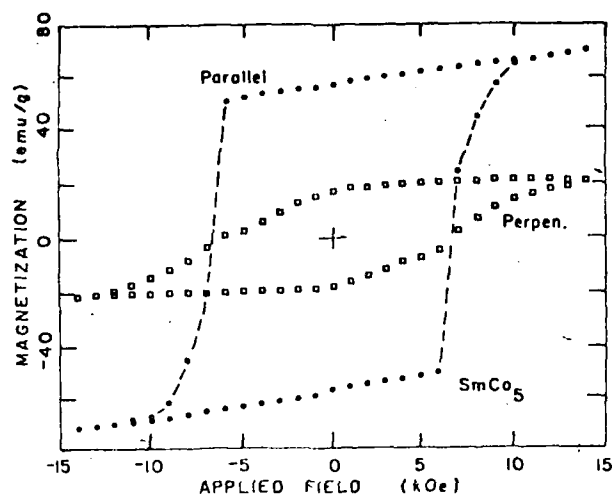


Fig. 7. Parallel to film plane and perpendicular hysteresis loops for a Sm-Co 1:5 sample synthesized onto 650 C substrate without any applied magnetic field during the sputtering, oxygen level = 1.4 at. %.

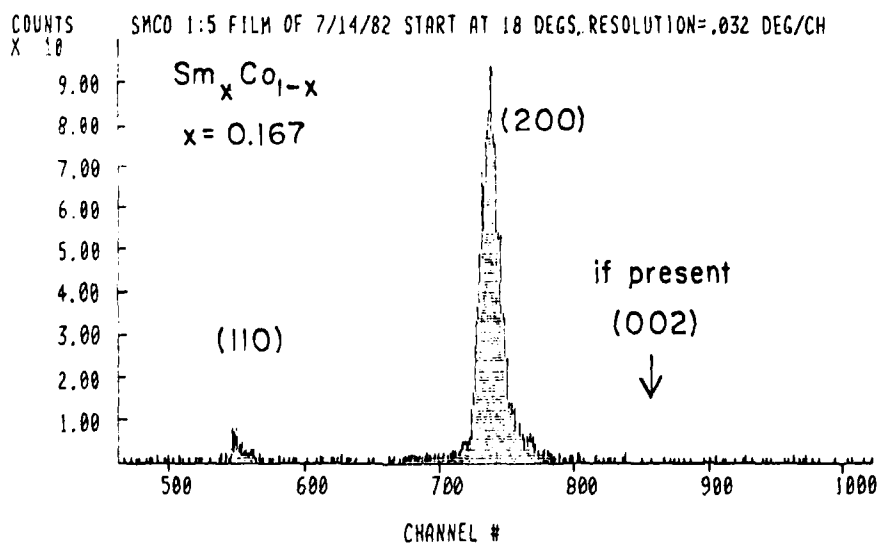


Fig. 8. X-Ray diffraction trace for the sample of Fig. 7. The (100) and (300) reflections are present off the ends of this trace. $I(200)/I(110) = 20:1$.

Table 1. Sm-Co 1:5 compound, T substrate = 600 C, P = 150 mTorr Ar,
1.75 kOe inplane field at 0 degrees during sputtering.

Inplanar Angle	M@14kOe (emu/g)	M rem/M 14 kOe	H coer. (kOe)	-H@ M/M rem = 0.9 (kOe)
0	67.5	0.966	8.9	5.5
45	67.5	0.969	7.6	5.3
90	65.2	0.962	7.5	5.5

In Fig. 10 and Fig. 11 are shown hysteresis loops for a sample synthesized onto a heated substrate in the presence of an inplane magnetic field of 1.75 kOe. This sample is slightly richer in Co than the stoichiometric composition and had a saturation moment of 114 emu/g when magnetized to an applied field of 150 kOe. The inplane loop in this case is flat topped and there is no appreciable change in slope upon entering the second quadrant. For an applied field of 14 kOe the maximum inplane moment is 76 emu/g for the case when the magnetometer field is parallel to the original field applied during sputtering, Fig. 10. The maximum moment for a field of 14 kOe applied perpendicular to the film plane is 16.6 emu/g. The ratio of the maximum inplane moment to the moment perpendicular for 14 kOe is 0.22. The magnetic hardness of this film is illustrated by the fact that the maximum inplane moment for 14 kOe applied inplane is only 0.67 of the moment for an applied inplane field of 150 kOe. This sample had an x-ray diffraction trace as shown in Fig. 12. The ratio of the (200) to the (110) intensity ratio was 10:1 which is consistent with an Auger analyzed

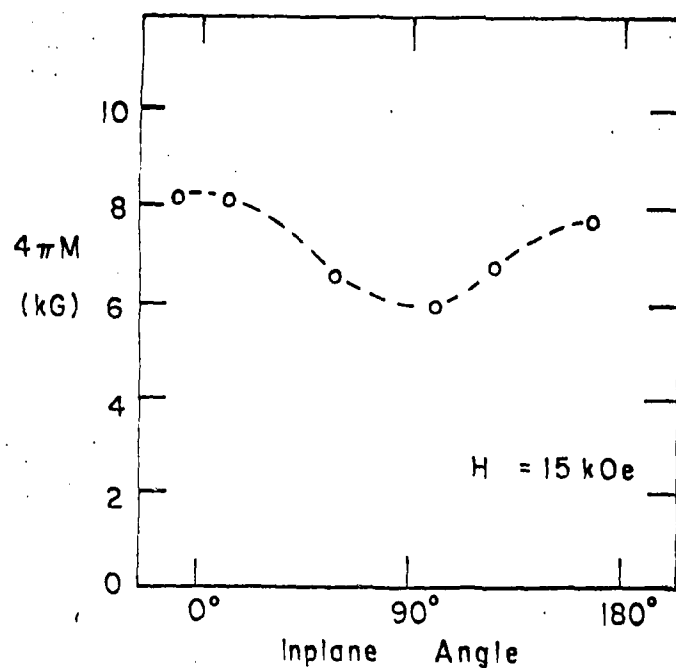


Fig. 9. Magnetization as a function of inplane angle for a magnetometer field of 15 kOe. During the sputter synthesis there was a field of 1.75 kOe applied inplane at 0 deg. reference angle.

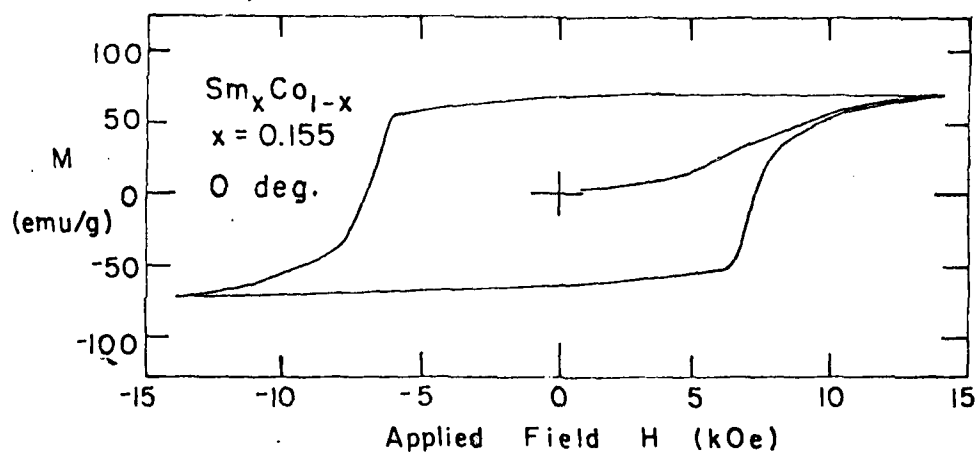


Fig. 10. Parallel to film plane in direction of field applied during sputtering hysteresis loop. This sample was sputtered with inplane field of 1.75 kOe at 0 deg.

oxygen content of 2.5 atomic %.

In Fig. 11 is shown the hysteresis loop for the sample of Fig. 10 but with the magnetometer field applied in the film plane but at 90 degrees to the field applied during sputtering. The loop is still square but the maximum moment reached is lower indicating some c-axis alignment has been achieved within the film plane. X-Ray measurements of the edge of this sample show the intensity ratio of the (003) reflection measured parallel to perpendicular of the sputtering field direction to be 1.126 ± 0.007 . This measurement was difficult and required long intervals of pulse counting to acquire sufficient statistics from the edge of a sample. The x-ray diffraction measurements indicate the c-axis to be rigidly aligned into the film plane and with 0.53:0.47 preferential alignment of the c-axis in the direction of the magnetic field applied during sputtering.

Films of the type shown in Fig. 10 for the Sm-Co system with 15.5 atomic % Sm have been measured to have a static energy product of 14 MG-Oe for a comparatively low initial magnetizing field of 14 kOe. Samples of this system for 16.7 atomic % Sm have been measured to have a static energy product of 10.5 MG-Oe for the same initial magnetizing field of 14 kOe.

The degree of inplane c-axis alignment has been correlated to an empirical equation for the initial magnetization as a function of inplane alignment fraction and inplane angle of the magnetometer field from the field direction applied during sputtering. The inplanar easy direction of magnetization is taken to be $A = A' \tanh(a + a^3) \cos(t)$ and the fraction of c-axis alignment as f . Here $a = H/H_0$ is the ratio of the applied

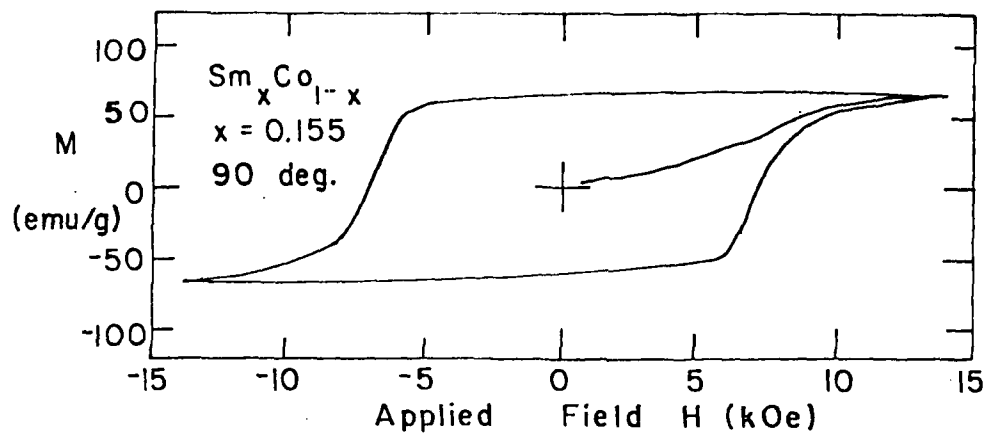


Fig. 11. Inplane hysteresis loop for sample of Fig. 10, but at 90 degrees to field applied during sputtering.

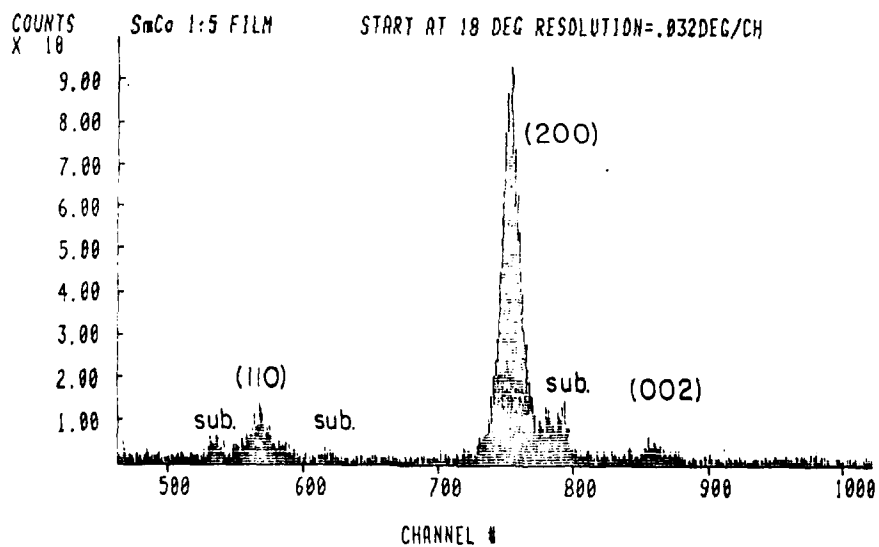


Fig. 12. X-Ray diffraction trace for sample of Fig. 10,11.

field to an effective coercive force. The inplanar hard direction magnetization is taken to be $B = B' \tanh(b) \sin(t)$ with an alignment fraction $(1 - f)$. The argument $b = H/H_a$ where H_a is the anisotropy field. The initial magnetization in the magnetometer field is then given by

$$M/M_s = [Af + B(1-f)](\cos(t))^2 + [A(1-f) + Bf](\sin(t))^2$$

where t is the inplanar angle between the magnetometer field and the field applied during sputtering. The value of f is then used as a fitting parameter to compare to the experimental magnetization value for the maximum applied fields. The best fits are obtained with $f = 0.53-0.55$ which is consistent with the alignment fraction as indicated from the x-ray analysis. The value of the anisotropy field H_a obtained from the magnetization measurements to 150 kOe for the sample of Fig. 10 is 87 kOe. Consequently the hard axis fraction contributes only slightly to the moment for applied fields in the order of 15 kOe. The square loop character is then a consequence of the near total remanence of the easy axis sample fraction. The variation of the inplanar magnetization shown in Fig. 9 is consistent with an inplanar alignment fraction of the easy axis, which in this case is the c-axis, of $f = 0.54$.

In summary, selectively thermalized trisputtering has been used to directly synthesise the Sm-Co 1:5 compound onto heated substrates in the presence of an applied inplane magnetic field of 1.75 kOe. The c-axis of the sputtered films is rigidly aligned into the substrate plane. The inplane field applied during sputtering has been used to achieve a partial preferential alignment of the c-axis within the film plane. Low oxygen

level samples sputtered with an applied inplane magnetic field have exhibited square flat topped hysteresis loops. Samples prepared onto heated substrates at $T = 585$ to 800 C show a correlation of film texture to oxygen content. For oxygen levels lower than 5 atomic % the (200) texture is dominant and for oxygen levels above 5 atomic % the (110) texture is dominant. No subsequent heat treatment or annealing has been necessary to obtain square flat topped hysteresis loops on the directly synthesized films.

Samples of the Sm-Fe system have also been trisputtered under conditions of selective thermalization. Three samples made with 1.4 atomic % oxygen (0.3 wt. % oxygen) showed only the 1-2, 1-3, and 2-17 compounds. This is in agreement with the work of Buschow(4) for the bulk pure Sm-Fe system. The various phases were observed along the length of each substrate and the results correlated with compositions as determined by x-ray fluorescence measurements. However, for a set of three Sm-Fe samples made with 6.5 atomic % oxygen (1.4 wt. % oxygen), four additional diffraction peaks were present at a composition corresponding to a Sm-Fe 1:5 phase. The observed reflections in this case correspond to (110), (111), (200), and (201). The lattice parameter was measured to be $a = 4.831 \pm 0.006$ Å and $c = 4.342 \pm 0.024$ Å. The ratio of the oxygen stabilized Sm-Fe 1:5 atomic volume to that of the Sm-Co 1:5 is 1.02. In contrast to the Sm-Co system, the larger moment for a magnetizing field of 14 kOe occurred when the magnetometer field was perpendicular to the film plane. In Fig. 13 is shown hysteresis loops for a parallel and perpendicular field for a sample with 17 atomic % Sm. The oxygen containing films that contained the 1:5 phase exhibited a maximum in the inplane

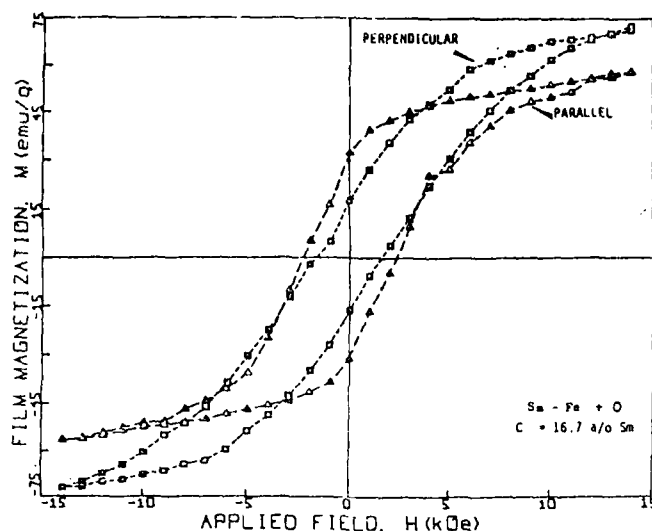


Fig. 13. Parallel to film plane and perpendicular to film plane hysteresis loops for Sm-Fe (16.7 at. % Sm) plus oxygen system sample. This composition would correspond to a 1:5 compound.

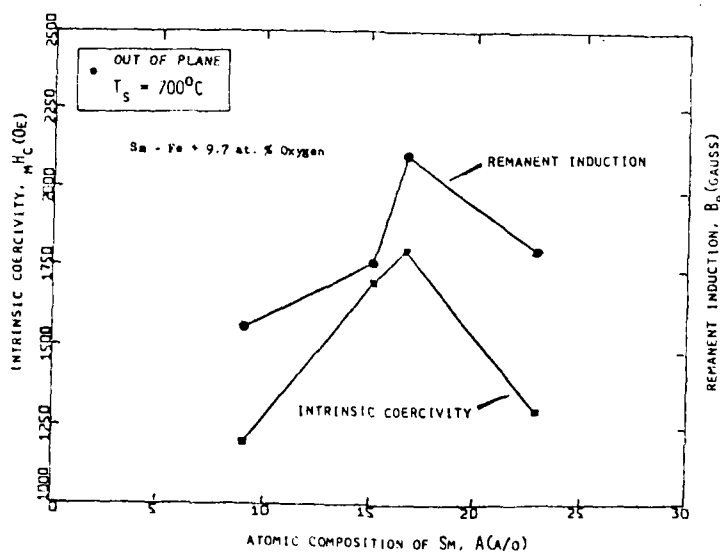


Fig. 14. Sm-Fe plus oxygen system showing maximum in remanent induction and coercivity at a composition corresponding to a 1:5 Sm-Fe metastable compound. The out of plane moment is larger than in plane moment for an applied field of 14 kOe. Magnetic measurements made at Grumman Aerospace Corp.

intrinsic coercive force of 2.4 kOe at 17 atomic % Sm. This maximum in the coercive force is illustrated in Fig. 14. The perpendicular intrinsic coercive force also exhibited a maximum of 1.9 kOe at this same composition. The low oxygen Sm-Fe samples did not exhibit the 1:5 diffraction lines and also did not exhibit a maximum in the coercive force in the vicinity of the 1-5 composition. For the low oxygen Sm-Fe samples, at a composition corresponding to 17 atomic % Sm, the intrinsic coercivity for an initial magnetizing field of 14 kOe was 1.5 kOe for an applied field parallel and 1.0 kOe for an applied field perpendicular to the film plane.

RESEARCH IN PROGRESS

Measurements are in progress to extend the x-ray and magnetic measurements to more Co rich compositions than the 1:5 phase. A large number of samples of this type have already been synthesized. The limiting factor in accumulating data has been that we have had to take samples to other locations to have magnetic measurements made. We have currently requested funds to purchase a vibrating sample magnetometer. A magnetometer facility has been built but it currently does not have sufficient sensitivity for magnetic measurements on small film samples. X-Ray diffraction analysis is proceeding on the samples which have been synthesized.

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